IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants	:	Simonsen et al.
Serial No.	:	10/611,795
Filed	:	June 30, 2003
Title	:	Stabilization of Granules
Examiner	:	Brian P. Mruk
Group Art unit	:	1751

DECLARATION OF OLE SIMONSEN, ERIK KJAER MARKUSSEN, HANNE ROJEL, SVEND KAASGAARD, THOMAS HONGER CALLISEN, CHRISTIAN ISAK JORGENSEN AND TOMAS TAGE HANSEN UNDER 37 C.F.R. 1.131

Commissioner for Patents
P.O.Box 1450
Alexandria, VA 22313-1450

Dear Sir:

We, Ole Simonsen, Erik Kjaer Markussen, Hanne Rojel, Svend Kaasgaard, Thomas Honger Callisen, Christian Isak Jorgensen, hereby declare and state as follows:

1. We, Ole Simonsen, Erik Kjaer Markussen, Hanne Rojel, Svend Kaasgaard, Thomas Honger Callisen, and Christian Isak Jorgensen, are the named inventors on the above-identified application ("the present application") and are informed as to the prosecution of the present application, particularly the present pending claims as set forth in Exhibit 1 attached hereto. Work reported in the present application was actually performed in Denmark, a GATT/TRIPS/WTO member country, prior to April 4, 2002.

2. For instance, attached as Exhibit 2 is a true and correct copy of an internal Novozymes A/S report [invention sheet] in English, prepared by the inventor Ole Simonsen in the ordinary course of business of his employment with Novozymes A/S and kept by him in the ordinary course of business, dated prior to April 4, 2002, reporting work of the named inventors done by inventor Ole Simonsen or under his direction, supervision or control, in the ordinary course of business of Novozymes A/S, prior to April 4, 2002. For example, the report parallels test examples in the present application

and provides, inter alia, a granule comprising a core matrix and one or more coatings, wherein the core matrix comprises: a) an active compound; b) a synthetic polymer in an amount of 0.1 to 10 % by weight of the core matrix; and c) antioxidant or reducing agent in an amount of 0.2 to 5 % by weight of the core matrix.; and a process for preparing a granule, comprising the steps of for preparing a granule, comprising the steps of:
a) preparing a core matrix comprising an active compound; a synthetic polymer in an amount of 0.1 to 10 % by weight of the core matrix; and antioxidant or reducing agent in an amount of 0.2 to 5 % by weight of the core matrix; and b) applying one or more coating to said core matrix.

3. I, Tomas Tage Hansen, can corroborate receipt of the report [invention sheet] before April 4, 2002. I am Director of Formulation Development at Novozymes A/S, and in the ordinary course of my employment with Novozymes A/S regularly receive, read and understand, in English, reports [invention sheets] from inventors, such as reports [Invention sheets] from inventor Ole Simonsen; and hereby confirm that I received, read and understood in English in the ordinary course of my employment with Novozymes A/S the report [Invention sheet] of Exhibit 2 prior to April 4, 2002.

4. Accordingly, embodiments of the invention were actually reduced to practice, prior to April 4, 2002, in Denmark, a GATT/TRIPS/WTO member country, and there was corroboration thereof of a person who was not an inventor, prior to April 4, 2002, in Denmark, a GATT/TRIPS/WTO member country.

5. We further declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and that these statements were made with the knowledge that willful, false statements and the like so made are punishable by fine or imprisonment, or both, under Title 18, section 1001, of the United States Code, and that such willful, false statements may jeopardize the validity of the application or any patent issuing thereon.

15/12/05
Date

Tomas Tage Hansen
Tomas Tage Hansen

14-DEC-2005
Date

Ole Simonsen

15-12-2005
Date

Erik Kjaer Markussen

15-12-2005
Date

Hanne Rojel

16-Dec-2005
Date

Svend Kaasgaard

13-Dec-2005
Date

Thomas Honger Callisen

14-Dec-2005
Date

Christian Isak Jorgensen

Exhibit 1

Pending Claims in U.S. Application No. 10/611,795

Claim 1. A granule comprising a core matrix and one or more coatings, wherein the core matrix comprises:

a. an active compound;

b. a synthetic polymer in an amount of 0.1 to 10 % by weight of the core matrix; and

c. antioxidant or reducing agent in an amount of 0.2 to 5 % by weight of the core matrix.

Claim 2. The granule according to claim 1, wherein the matrix further comprises a polysaccharide in an amount greater than 2 % by weight of the core matrix.

Claim 3. The granule according to claim 1, wherein the synthetic polymer is present in an amount of 1 to 2 % by weight of the core matrix.

Claim 4. The granule according to claim 1, wherein the antioxidant or reducing agent are present in an amount of 1 to 3 % by weight of the core matrix.

Claim 5. The granule according to claim 1, wherein the active compound is an enzyme.

Claim 6. The granule according to claim 1, wherein the synthetic polymer is a polyvinyl polymer selected from the group consisting of PVP, PVA and copolymers thereof.

Claim 7. The granule according to claim 1, wherein the antioxidant or reducing agent is selected from the group of sodium thiosulfate, sodium sulfite, thiodipropionic acid, erythorbate, ascorbate or methionine.

Claim 8. The granule according to claim 1, wherein the synthetic polymer is PVP and the antioxidant is sodium thiosulfate.

Claim 9. The granule according to claim 2, wherein the amount of polysaccharide in the core matrix is 2 to 75 % by weight of the core matrix.

Claim 10. The granule according to claim 2, wherein the polysaccharide is starch.

Claim 11. The granule according to claim 1, where the core matrix is coated onto a preformed core.

Claim 12. The granule of claim 1, further comprising Magnesium sulfate or hydrated magnesium sulfate.

Claim 13. The granule according to claim 12, wherein the magnesium sulfate is present in an amount of 1 to 70 % by weight of the core matrix.

Claim 14. The granule according to claim 1, wherein the granule is coated with a salt layer.

Claim 15. The granule according to claim 14, wherein the salt layer contains 2% to 30% by weight of the core matrix and salt layer.

Claim 16. The granule according to claim 14, wherein the salt layer contains 3 to 10 % by weight of the core matrix and the salt layer.

Claim 17. The granule according to claim 14, wherein the salt layer is 2 to 100 µ thick.

Claim 18. The granule according to claim 1, wherein the granule further comprises a protective coating.

Claim 19. A process for preparing a granule, comprising the steps of:

a. preparing a core matrix comprising an active compound; a synthetic polymer in an amount of 0.1 to 10 % by weight of the core matrix; and antioxidant or reducing agent in an amount of 0.2 to 5 % by weight of the core matrix;

b. and applying one or more coating to said core matrix.

Claim 20. The process according to claim 19, where the granules are prepared in a mixer, a fluid bed, a fluid bed spray dryer, a spray dryer or an extruder.

Novozymes Patents

INVENTION SHEET
NOVOZYMES A/S
- Strictly confidential-

This sheet must be filled-in as soon as possible after a potentially patentable invention has been conceived. The sheet should immediately be sent to the responsible Project Leader (PL) and Director (DIR), contact person in Patents (Pat-X), GeK/PDSh or ELam, IPP-DIR and L&S-MGR by email, and a signed and corroborated copy must be sent to GeK/PDSh or ELam/RLS by mail.

To be filled in by the Inventor:

1. **Suggested Inventors (initials or name):**
   OSi, EMa, HanR, SGK, Call, ClsJ

2. **Suggestion for composition of the Working Group (WG), i.e. persons to be involved in drafting and prosecuting the patent application:**
   OSi, EMa, TTH

3. **Project closest related to the invention; Responsible PL and DIR:**
   BesT, BesT implementation (PL: MeTF)

4. **Brief description of the invention:**
   The quality of our enzyme granulates can be improved significantly by addition of the following components in the enzyme matrix/layer:
   - Polymer (e.g. PVP, PVP copolymers) (0.2-5% pref. 1-2%)
   - Organic matter (e.g. starch (rice, corn), protein, flour, sludge from e.g. enzyme fermentation/recovery) (2-50%, pref. 5-20%)
   - Antioxidant (e.g. Sodium thiosulfate) (0.2-5%, pref. 1-3%)

   The combination of these three is optimal

   The technology can be implemented for all types of enzyme granules (high shear granules (T-granulates), core granulates (TK and GCI type), extrudates)

   The components especially increase storage stability in powder detergents, both bleach and non-bleach and at low and high humidity. This effect can be further improved by:
   - Addition of $MgSO_4$ (2-60%)
   - Spray-drying the enzyme with one or more of the stabilizers (incl. $MgSO_4$), typically the enzyme constitute around 50% of the spray-dried powder)
   - Addition of thin salt coating layer

   The granulates have also proved to be superior to current T-granules with regard to:

   - Organic matter (especially starch) reduces odour from e.g. thiosulfate
   - Particle size can be reduced without increasing dust
   - Density is reduced
   - More homogeneous enzyme activity (activity as function of particle size)
   - Less dust
   - More spherical shape

REDACTED

- Increased whiteness (reduced titan consumption)

Other relevant issues:
It is the combination of the stabilizers that gives the overall increased quality. Example: Thiosulfate is an efficient antioxidant, but it is also hygroscopic, so if the amount is increased above a certain level (around 3%) the negative effect from the hygroscopic nature is getting dominating. PVP reduces the negative effect of thiosulfate and a further bleach stability can be obtained by inclusion of starch.

5. **Potential utility and advantages of the invention:**
The improvements can readily and relatively cheap be implemented for our T-granulates, and the implementation has already been initiated

6. **Brief description of the experimental work done to date:**
A relatively huge data material with different types and amounts of the components have been prepared, and is being prepared currently

7. **Any external agreements or collaborations to be taken into account:**
None

8. **Please state any relevant background art and/or result of a novelty search:**
NZ and GCI granulate patents in general, EP-075818A1 (spray-drying)
May be combined with the antioxidant/slurry patent currently being prepared

9. **Expected commercial relevance for Novozymes A/S:**
Very commercially relevant for the detergent industry (and on relatively short term, i.e. within 6-9 months).

10. **Any special reasons for urgency:**
Se above

11. **This Invention Sheet is submitted by (Initials or name, phone, fax):**
OSi, 26257

Date: Signature of Inventor:

Date: Corroboration signature:

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Completion of
INVENTION SHEET

To be filled in by the Patent Attorney (and forwarded to WG, PPG, RG, L&S-MGR, IPP-DIR, GeK/PDSh, ELam, RLS and secretary)

Docket number: #
Attorney/Secretary (Pat-X): #
PPG: #
ISG: #
Action:

☐ 1) An application is to be filed

a) **Suggestions as to type and scope of claims (for filing and novelty search)**

b) **Novelty search: Responsibility and target date**

c) **Examples and/or background to be included: responsibility and target date**

d) **First draft of the claims/application to WG or comment: target date**

e) **Final draft for approval by external partners (if any): target date**

f) **Expected filing date**

☐ 2) ON HOLD (further experimental work is needed before patenting is decided)

Key person:
Action due date:

Comments:

☐ 3) Other types of actions

a) Prophylactic publication of this know-how is suggested;

Comments:

b) This know-how is suggested not to be patented, but to be kept secret;

Comments:

Date: # Signature of Patent Attorney:

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